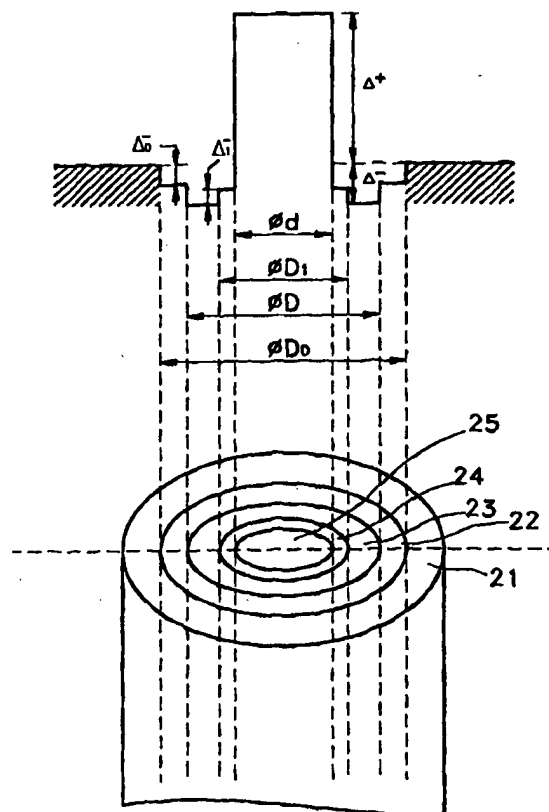


## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/KR99/00329 <b>(22) International Filing Date:</b> 22 June 1999 (22.06.99)  <b>(30) Priority Data:</b> 1998/24159                      25 June 1998 (25.06.98)                      KR 1999/2696                        28 January 1999 (28.01.99)                      KR  <b>(71) Applicant:</b> SAMSUNG ELECTRONICS CO., LTD. [KR/KR]; 416, Maetan-dong, Paldal-gu, Suwon-city, Kyungki-do 442-373 (KR).  <b>(72) Inventors:</b> OH, Sung, Koog; 4-504 Samsung Sawon Apt., 109-1, Gongdan 1-dong, Gumi-city, Kyungsangbuk-do 730-031 (KR). SEO, Man-Seok; 103-904 Daeback Apt., Okgae-dong, Gumi-city, Kyungsangbuk-do 730-380 (KR). DO, Mun-Hyun; 9-505 Samsung Apt., 37, Songjung-dong, Gumi-city, Kyungsangbuk-do 730-090 (KR). YANG, Jin-Seong; 3-201 Samsung Sawon Apt., Gongdan 1-dong, Gumi-city, Kyungsangbuk-do 730-031 (KR).  <b>(74) Agent:</b> LEE, Young, Pil; The Cheonghwa Building, 1571-18, Seocho-dong, Seocho-gu, Seoul 137-073 (KR).		<b>(81) Designated States:</b> AU, BR, CA, CN, IN, JP, MX, RU, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.</i> <i>Before the expiration of the time limit for amending the</i> <i>claims and to be republished in the event of the receipt of</i> <i>amendments.</i>
<b>(54) Title:</b> OPTICAL FIBER PREFORM HAVING OH BARRIER AND MANUFACTURING METHOD THEREOF		
<b>(57) Abstract</b> <p>An optical fiber preform having a substrate tube, a cladding layer and a core layer further includes a first barrier layer deposited by a material having a low OH diffusion coefficient between the substrate tube and the cladding layer, wherein the first barrier layer is for substantially preventing OH contained in the substrate tube from being diffused into the cladding layer. The optical fiber preform further includes a second barrier layer formed by depositing a material having a low OH diffusion coefficient between the cladding layer and core layer, for substantially preventing OH which has been diffused into the cladding layer from the substrate tube from being diffused further into the core layer. Outer and inner OH barriers containing no P<sub>2</sub>O<sub>5</sub> are deposited between the substrate tube and the cladding layer and between the cladding layer and the core layer in a deposition process, such that OH can be effectively prevented from being diffused from the substrate tube to the core layer in a core deposition process, a collapsing process or a closing process.</p>		



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## OPTICAL FIBER PREFORM HAVING OH BARRIER AND MANUFACTURING METHOD THEREOF

### 5    Technical Field

The present invention relates to a general optical fiber preform, and more particularly, to an optical fiber preform for minimizing the diffusion of OH from the substrate tube to the core of an optical fiber, and a manufacturing method thereof.

### 10   Background Art

A single mode optical fiber is made by depositing a cladding layer and a core layer. In a DC-SM (depressed cladding-single mode) type, a cladding layer is deposited by doping  $\text{SiO}_2$  with  $\text{P}_2\text{O}_5$ ,  $\text{GeO}_2$ , and F to lower the deposition temperature and the refractive index, a core layer for transmitting light is deposited  
15 by doping  $\text{SiO}_2$  with  $\text{GeO}_2$  to increase the refractive index, and then an optical fiber preform is manufactured through a collapsing and closing process.

In a process for manufacturing an optical fiber preform using modified chemical vapor deposition (MCVD), self-collapse of a substrate tube occurs during deposition as the deposition layer becomes thicker, resulting in an increase in the  
20 thickness of the tube. Also, a high temperature burner is required to sinter and consolidate a thick deposition layer, and the time for the collapsing and closing process becomes longer, so that a substrate tube becomes exposed to a high temperature over a long period of time.

In this process, while a very small amount of water ( $\text{H}_2\text{O}$ ) (generally about  
25 several ppm) contained in the substrate tube is diffused into the deposition layer, diffused water is combined with  $\text{P}_2\text{O}_5$  or  $\text{SiO}_2$  deposited in the cladding region, thus forming a P-O-H or Ge-O-H bond combination. OH diffused up to the core region is combined with  $\text{SiO}_2$  or  $\text{GeO}_2$  deposited in the core layer, thus forming an Si-O-H or Ge-O-H bond combination while dissolving Si-O or Ge-O bond  
30 combination.

O-H or P-O-H bond combination formed in combination with water in each deposition region as described above results in additional optical loss due to an

absorption band at a specific wavelength region. In the case of a single mode optical fiber, wavelength bands in which serious optical loss occurs are a  $1.24\mu\text{m}$ - $1.385\mu\text{m}$  band due to the O-H bond combination, and a  $1.2$ - $1.8\mu\text{m}$  band due to the P-O-H bond combination. When OH is diffused into the core region, it forms a  
5 non-bridging oxygen (NBO), and the structural homogeneity of glass material of the core layer is thus locally deteriorated, which causes density fluctuation of the core layer. Consequently, scattering loss is increased.

The inside and outside diameters of a tube contract with an increase in the thickness of the deposition layer during sintering performed simultaneously with  
10 deposition, so that it is difficult to obtain an appropriate diameter ratio (that is, cladding diameter/core diameter =  $D/d$ ). Therefore, a distance sufficient to prevent diffusion of OH cannot be secured, thus greatly increasing loss due to OH.

In the prior art, a method of thickening the cladding layer is used to prevent OH from diffusing from the substrate tube to the core layer. However, when a  
15 large-aperture preform is manufactured by this method, contraction of a tube makes it difficult to secure an appropriate diameter ratio, a burner of a higher temperature is required during deposition of the core layer since the efficiency of transmitting heat to a core layer is degraded due to an increase in the thickness of the tube layer. Thus, the tube is exposed to high temperature for a long time, thus  
20 increasing loss due to OH.

### Disclosure of the Invention

To solve the above problems, it is an objective of the present invention to provide an optical fiber preform capable of effectively reducing loss due to OH  
25 while lowering the diameter ratio by forming a barrier layer for blocking or remarkably alleviating diffusion of OH between a substrate tube and a core layer in order to prevent OH from diffusing from the substrate tube into the core layer.

It is another objective of the present invention to provide a method of manufacturing an optical fiber preform having an OH barrier.

30 Accordingly, to achieve the first objective, there is provided an optical fiber preform having a substrate tube, a cladding layer and a core layer, the optical fiber preform further comprising a first barrier layer deposited by a material having a

low OH diffusion coefficient between the substrate tube and the cladding layer, wherein the first barrier layer is for substantially preventing OH contained in the substrate tube from being diffused into the cladding layer.

It is preferable that the optical fiber preform further comprises a second  
5 barrier layer formed by depositing a material having a low OH diffusion coefficient between the cladding layer and core layer, for substantially preventing OH which has been diffused into the cladding layer from the substrate tube from being diffused further into the core layer.

To achieve the first objective, there is provided another optical fiber  
10 preform having a substrate tube, a cladding layer and a core layer, the optical fiber preform further comprising a first barrier layer deposited by a material having a low OH diffusion coefficient between the substrate tube and the cladding layer, wherein the first barrier layer is for substantially preventing OH contained in the substrate tube from being diffused into the cladding layer, wherein the refractive  
15 index of the core layer is greater than the refractive index of the cladding layer and gradually increases in the direction from the outside of the core layer to the center of the core layer.

It is preferable that this optical fiber preform further comprises a second  
20 barrier layer deposited by a material having a low OH diffusion coefficient between the cladding layer and core layer, wherein the second barrier layer is for substantially preventing OH diffused into the cladding layer from being diffused further into the core layer.

To achieve the second objective, there is provided a method of  
manufacturing an optical fiber preform having a substrate tube, a cladding layer and  
25 a core layer, the method comprising the steps of: forming a first barrier layer by depositing a material having a low OH diffusion coefficient; forming a cladding layer by doping a material suitable for lowering a process temperature and increasing deposition efficiency; and forming a core layer being a region through which an optical signal is transmitted.

30 It is preferable that a second barrier layer is further formed by depositing a material having a low OH diffusion coefficient, before the core layer is formed after the cladding layer is formed. Also, it is preferable that the core layer is

formed so that the refractive index gradually increases in the direction from the outside to the center of the core layer.

#### Brief Description of the Drawings

- 5           FIG. 1 is a view illustrating a general single mode optical fiber;  
            FIG. 2 is a view illustrating a single mode optical fiber according to the present invention;  
            FIG. 3 is a view illustrating another single mode optical fiber according to the present invention; and  
10          FIG. 4A, 4B and 4C are views illustrating a method of manufacturing a signal mode optical fiber according to the present invention using a modified chemical vapor deposition (MCVD) method.

#### Best mode for carrying out the Invention

- 15          Preferred embodiments of the present invention will now be described in more detail with reference to the attached drawings.

Referring to FIG. 1 showing a general depressed cladding-single mode (DC-SM) optical fiber, reference numeral 11 denotes a substrate tube, reference numeral 12 denotes a cladding layer, and reference numeral 13 denotes a core layer. Also,  
20    $\Delta^+$  represents the refractive index of the core layer and  $\Delta^-$  represents the refractive index of the cladding layer, relative to the refractive index of the substrate tube, respectively. Also,  $\Phi d$  represents the diameter of the core layer, and  $\Phi D$  represents the diameter of the cladding layer.

$P_2O_5$  is deposited to form the cladding layer 12.  $P_2O_5$  has a relatively low  
25   melting point of about 570°C. so when it is used together with a different source material, the process temperature can be lowered and deposition efficiency can be increased. On the other hand, since the  $P_2O_5$  doped on the cladding layer 12 has a large hygroscopicity, it acts as an OH bridge for transmitting OH contained in the substrate tube 11 to the core layer 13. Therefore, loss due to OH in the core layer  
30   13 is increased.

FIG. 2 is a view illustrating a single mode optical fiber according to the present invention. In FIG. 2, reference numeral 21 denotes a substrate tube,

reference numeral 22 denotes a first barrier layer (outer cladding layer), reference numeral 23 denotes a middle cladding layer, reference numeral 24 denotes a second barrier layer (inner cladding layer), and reference numeral 25 denotes a core layer. Also,  $\Delta^+$  represents the relative refractive index of the core layer 25, and  $\Delta$  represents the refractive index of the middle cladding layer 23, which are relative indices to that of the substrate tube 21.  $\Delta_0^-$  represents the refractive index of the first barrier layer 22, and  $\Delta_1^-$  represents the refractive index of the second barrier layer 24, which are relative indices to that of the middle barrier layer 23.  $\Phi d$  represents the diameter of the core layer 25.  $\Phi D_1$  represents the diameter of the second barrier layer 24,  $\Phi D$  represents the diameter of the middle cladding layer 23, and  $\Phi D_0$  represents the diameter of the first barrier layer 22.

As described above, the cladding layer of the optical fiber preform according to the present invention is comprised of three layers each having a different chemical composition rate. In other words, the cladding layer is comprised of the first barrier layer (outer cladding layer) 22, the middle cladding layer 23, and the second barrier layer (inner cladding layer) 24.

The first barrier layer (outer cladding layer) 22 is positioned between the substrate tube 21 having a large OH concentration and the middle cladding layer 23 containing the OH carrier  $P_2O_5$ , and prevents OH contained in the substrate tube 21 from being diffused into the middle cladding layer 23. The second barrier layer (inner cladding layer) 24 is positioned between the middle cladding layer 23 and the core layer 25, and prevents OH diffused from the substrate tube 21 into the middle cladding layer 23 in spite of the first barrier layer 22 from further penetrating into the core layer 25. The first and second barrier layers 22 and 24 do not contain  $P_2O_5$  which acts as an OH bridge, the refractive indices thereof are controlled using  $SiO_2$ ,  $GeO_2$ , and F, and the thicknesses thereof are appropriately controlled according to the overall thickness of the cladding layer. In particular, only the first barrier layer 22 can be interposed between the substrate tube 21 having a large concentration of OH and the middle cladding layer 23, or only the second barrier layer 24 can be interposed between the middle cladding layer 23 and the core layer 25.

Referring to the refractive index characteristics of the optical fiber preform,

the refractive index of the core layer 25 is greater than that of the cladding layers 22, 23 and 24. Thus, the refractive index of each of the outer and inner cladding layers 22 and 24 is controlled to be the same as or similar to the refractive index of the middle cladding layer 23. Also, the refractive indices of these three layers  
5 can be controlled to be the same.

In general, the concentration of OH in the deposition layer is about 1/1000 or less of the concentration of OH in the substrate tube. However, the cladding layer is deposited by doping  $P_2O_5$  in order to lower the process temperature in the cladding deposition process. Here, the  $P_2O_5$  has a large hygroscopicity.  
10 Accordingly, the  $P_2O_5$  deposited in the cladding layer acts as a bridge for transmitting OH from the substrate tube to the core layer, thus increasing loss due to OH in the core layer. Hence, in the present invention, an OH barrier doped with materials having low OH diffusion coefficients is formed between the substrate tube having a large concentration of OH and the cladding layer containing the OH  
15 carrier  $P_2O_5$ , or/and between the cladding layer and the core layer. The thus-formed OH barrier can prevent the diffusion of OH from the substrate tube 21 to the core layer 25.

FIG. 3 is a view illustrating another single mode optical fiber according to the present invention. In FIG. 3, reference numeral 31 denotes a substrate tube,  
20 reference numeral 34 denotes a first barrier layer (outer cladding layer), reference numeral 32 denotes a middle cladding layer, reference numeral 35 denotes a second barrier layer (inner cladding layer), and reference numeral 33 denotes a core layer. Also,  $\Delta N^+$  represents the refractive index of the core layer 33, and  $\Delta N^-$  represents the refractive index of the middle cladding layer 32, which are relative indices to  
25 that of the substrate tube 31.

As described above, the cladding layer of the optical fiber preform according to the present invention is comprised of three layers each having a different chemical composition rate. In other words, the cladding layer is comprised of the first barrier layer (outer cladding layer) 34, the middle cladding  
30 layer 32, and the second barrier layer (inner cladding layer) 35.

The first barrier layer (outer cladding layer) 34 is positioned between the substrate tube 31 having a large OH concentration and the middle cladding layer



32 containing the OH carrier  $P_2O_5$ , and prevents OH contained in the substrate tube 31 from being diffused into the middle cladding layer 32. The second barrier layer (inner cladding layer ) 35 is positioned between the middle cladding layer 32 and the core layer 33, and prevents OH diffused from the substrate tube 31 into the middle cladding layer 32 or OH resulting from water contained in a chemical material during deposition of the middle cladding layer 32, from penetrating into the core layer 33 which is an optical waveguiding region. The refractive index of each of the outer and inner cladding layers 34 and 35 is controlled to be the same as or similar to the refractive index of the middle cladding layer 32, and not to be greater than the refractive index of the substrate tube 31 or core layer 33.

The amount of OH contained in the substrate tube is relatively high compared to that of silica for deposition. Silica is the most stable deposition chemical material against an OH component in structure and can effectively block the diffusion of OH at a high temperature. Hence, the first and second barrier layers 34 and 35 do not contain  $P_2O_5$  acting as an OH bridge, the refractive index of the cladding is controlled using  $SiO_2$ , Ge, or F, and the thicknesses of these barrier layers are appropriately controlled according to the overall thickness of the cladding layer.

Referring to the refractive index characteristics of the optical fiber preform, the refractive index of the core layer 33 is greater than that of the cladding layers 32, 34 and 35, and the refractive index of the core layer 33 increases at a constant rate toward the center of the core layer. Thermal stress due to quick freezing is generated when an optical fiber is drawn out from the preform at high speed. Accordingly, the refractive index of the core layer 33 gradually increases from the refractive index  $\Delta N_0$  of the boundary toward the center thereof, thereby finally making the refractive index  $\Delta N$  at the center the greatest. By doing this, the optical loss of the optical fiber due to thermal stress, and degradation of the mechanical characteristics of the optical fiber can be prevented, and thus an optical fiber having a low loss and a low diameter ratio can be drawn out at high speed. For example, it is preferable that the refractive index of the outermost portion of the core layer is 75 to 99% of that of the center of the core layer.

FIGS. 4A, 4B and 4C are views illustrating a method of manufacturing the

single mode optical fiber according to the present invention shown in FIG. 2 or 3 using a modified chemical vapor deposition (MCVD) method. In the MCVD method, high purity carrier gases such as  $\text{SiCl}_4$ ,  $\text{GeCl}_4$ ,  $\text{POCl}_3$ , or  $\text{BCl}_3$  are introduced together with oxygen into a substrate tube 41 made of glass, and heat is then applied to the substrate tube 41 by a heating means 43, whereby soot, an oxidized deposit, is formed on the inside of the substrate tube by thermal oxidation, in FIG. 4A. Here, the concentration of the source gas is accurately controlled by a computer to adjust the refractive index, thereby depositing a cladding layer/core layer 42. The heating means 43 applies heat to the substrate tube 41 which rotates in the direction indicated by a rotating arrow, while the heating means moves in the direction indicated by the straight arrow. The source gases to be deposited are introduced into the substrate tube 41 through an inlet connected to a source material storage unit. A mixing valve and a blocking valve measure the flow of the source materials introduced into the substrate tube and perform adjustments necessary for mixture of the source materials.

In a process for depositing a cladding layer in the present invention, first, an outer cladding layer (a first barrier) is formed by depositing a material having a low OH diffusion coefficient excluding an OH carrier material such as  $\text{P}_2\text{O}_5$  having a large hygroscopicity. Another material suitable for lowering the process temperature and increasing deposition efficiency is doped, thereby forming a middle cladding layer. A material having a low OH diffusion coefficient is deposited excluding an OH carrier material such as  $\text{P}_2\text{O}_5$ , thereby forming an inner cladding layer (a second barrier). A core layer, a region where an optical signal is transmitted, is then formed. Therefore, the mixing of source gases introduced into the substrate tube 41 becomes different according to each deposition layer, and this mixing can be accomplished by appropriately controlling the mixing valve and the blocking valve.

In a process for depositing the core layer, the core layer is deposited so that the refractive index is constant from the outside to the center thereof, or so that the refractive index gradually increases in the direction from the outside to the center thereof.

FIG. 4B shows a cladding layer/core layer 40 deposited within the substrate

tube 41. In FIG. 4B, reference numeral 43 denotes an outer cladding layer, reference numeral 44 denotes a middle cladding layer, reference numeral 45 denotes an inner cladding layer, and reference numeral 46 denotes a core layer.

Referring to FIG. 4C, the deposited layers as shown in FIG. 4B are  
5 collapsed and closed by applying heat to the substrate tube 41, on which the cladding layer/core layer 40 has been deposited, using the heating means 43, thereby forming an optical fiber preform 47.

In a deposition process, the outer and inner OH barriers 43 and 45, which have the middle cladding layer 44 therebetween and do not contain  $P_2O_5$  acting as  
10 an OH bridge, are deposited, thereby effectively preventing OH from being diffused from the substrate tube 41 into the core layer 46 during a core deposition process, a collapsing process or a closing process. Accordingly, the loss due to an OH absorption band in the core layer can be minimized while an appropriate diameter ratio ( $D/d$ ) is maintained. Also, the diameter ratio can be made small,  
15 and thus the frequency of deposition can be reduced, thereby shortening the processing time. Here, it is preferable that a ratio ( $D/d$ ) of the diameter ( $D$ ) of the cladding layer to the diameter ( $d$ ) of the core layer is 1.1 to 3.0.

Meanwhile, in a sintering process performed simultaneously with deposition, self-collapse due to internal surface tension occurs in a process for sintering and  
20 consolidating soot particles. A buffer layer having a similar viscosity to the substrate tube exists between the substrate tube having a high viscosity and the cladding layer having a relatively low viscosity, such that the deterrent power of the tube is improved, and contraction of the tube can thus be reduced.

When an optical fiber preform is manufactured using the MCVD method,  
25 the total processing time becomes shorter as the diameter ratio becomes smaller, and a small diameter ratio is very favorable to the manufacture of a preform having a large aperture. In the prior art, when a diameter ratio becomes small, the OH loss is suddenly increased, thus deteriorating the quality of an optical fiber. Thus, it is commonly known that the diameter ratio is about 3.0. However, according to  
30 the present invention, even when the diameter ratio is reduced to less than 3.0, for example, to about 1.1 to 3.0, the OH absorption loss can be reduced, and loss due to thermal stress can also be minimized.

Industrial Applicability

In the present invention, according to optical fiber preforms having an OH barrier and a manufacturing method thereof as described above, outer and inner OH barriers containing no  $P_2O_5$  are deposited between a substrate tube and a cladding layer and between the cladding layer and a core layer in a deposition process, such that OH is effectively prevented from being diffused from the substrate tube to the core layer in a core deposition process, a collapsing process or a closing process. Hence, loss due to OH in the core layer can be prevented. Also, the core layer is formed to increase its refractive index in the direction from the outside to the center, such that degradation of characteristics due to high-speed drawing-out of an optical fiber from the preform can be prevented.

What is claimed is:

1. An optical fiber preform having a substrate tube, a cladding layer and a core layer, the optical fiber preform further comprising a first barrier layer deposited by a material having a low OH diffusion coefficient between the substrate  
5 tube and the cladding layer, wherein the first barrier layer is for substantially preventing OH contained in the substrate tube from being diffused into the cladding layer.
2. The optical fiber preform of claim 1, further comprising a second  
10 barrier layer formed by depositing a material having a low OH diffusion coefficient between the cladding layer and core layer, for substantially preventing OH which has been diffused into the cladding layer from the substrate tube from being diffused further into the core layer.
- 15 3. The optical fiber preform of claim 1 or 2, wherein the refractive index of the first or second barrier layer is controlled by doping  $\text{SiO}_2$ ,  $\text{GeO}_2$ , or F, and does not contain  $\text{P}_2\text{O}_5$ .
4. The optical fiber preform of claim 2, wherein the refractive index  
20 of the first or second barrier layer is controlled to be the same as or greater than the refractive index of the cladding layer.
5. The optical fiber preform of claim 1, wherein the ratio (D/d) of the diameter (D) of the cladding layer to the diameter (d) of the core layer is 1.1 to  
25 3.0.
6. An optical fiber preform having a substrate tube, a cladding layer and a core layer, the optical fiber preform further comprising a first barrier layer deposited by a material having a low OH diffusion coefficient between the substrate  
30 tube and the cladding layer, wherein the first barrier layer is for substantially preventing OH contained in the substrate tube from being diffused into the cladding layer, wherein the refractive index of the core layer is greater than the refractive

index of the cladding layer and gradually increases in the direction from the outside of the core layer to the center of the core layer.

7. The optical fiber preform of claim 6, further comprising a second  
5 barrier layer deposited by a material having a low OH diffusion coefficient between the cladding layer and core layer, wherein the second barrier layer is for substantially preventing OH diffused into the cladding layer from being diffused further into the core layer.

10 8. The optical fiber preform of claim 6 or 7, wherein the refractive index of the first or second barrier layer is controlled by doping  $\text{SiO}_2$ ,  $\text{GeO}_2$ , or F, and does not contain  $\text{P}_2\text{O}_5$ .

9. The optical fiber preform of claim 6 or 7, wherein the refractive  
15 index of the first or second barrier layer is controlled to be the same as or greater than the refractive index of the cladding layer.

10. The optical fiber preform of claim 6, wherein the ratio (D/d) of the  
20 diameter (D) of the cladding layer to the diameter (d) of the core layer is 1.1 to 3.0.

11. The optical fiber preform of claim 6, wherein the refractive index  
at the outermost point of the core layer is 75 to 99% of the refractive index at the  
center of the core layer.

25

12. A method of manufacturing an optical fiber preform having a  
substrate tube, a cladding layer and a core layer, the method comprising the steps  
of:

forming a first barrier layer by depositing a material having a low OH  
30 diffusion coefficient:

forming a cladding layer by doping a material suitable for lowering a  
process temperature and increasing deposition efficiency; and

forming a core layer being a region through which an optical signal is transmitted.

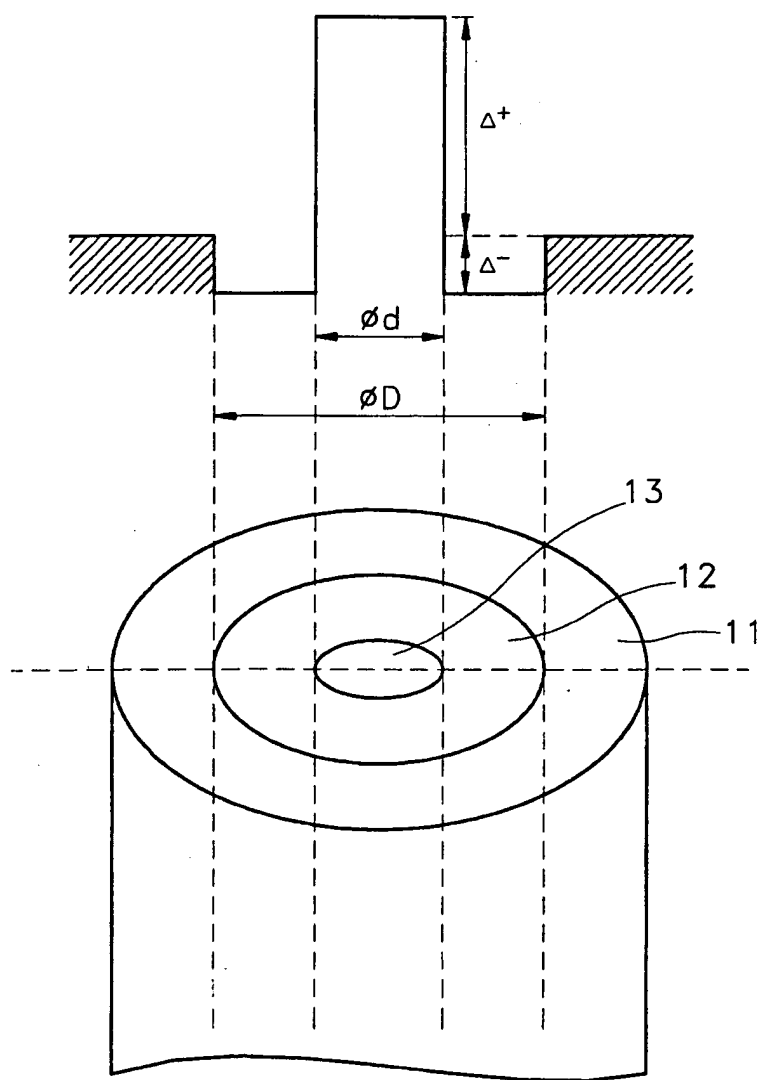
13. The method of claim 12, wherein a second barrier layer is further  
5 formed by depositing a material having a low OH diffusion coefficient, before the  
core layer is formed after the cladding layer is formed.

14. The method of claim 12, wherein the core layer is formed so that the  
refractive index gradually increases in the direction from the outside to the center  
10 of the core layer.

15. The method of claim 12 or 13, wherein the refractive index of the  
first or second barrier layer is controlled by doping  $SiO_2$ ,  $GeO_2$ , or F, and does  
not contain  $P_2O_5$  having a relatively large hygroscopicity.

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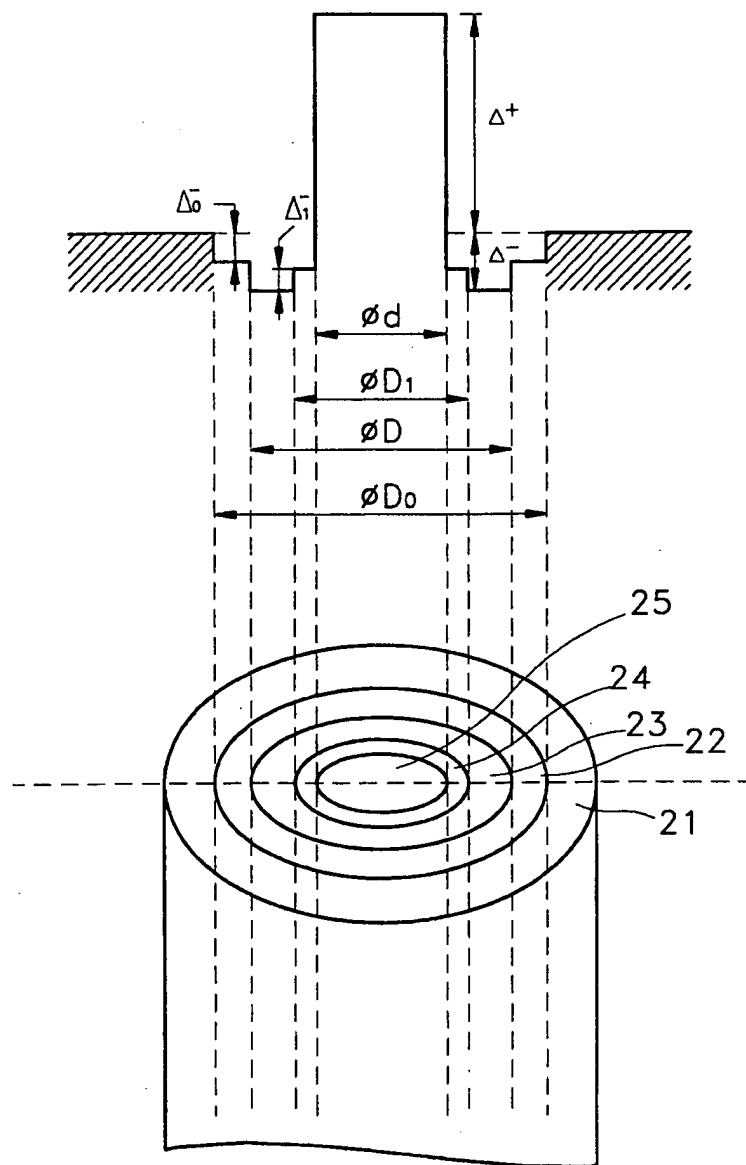
FIG. 1 (PRIOR ART)





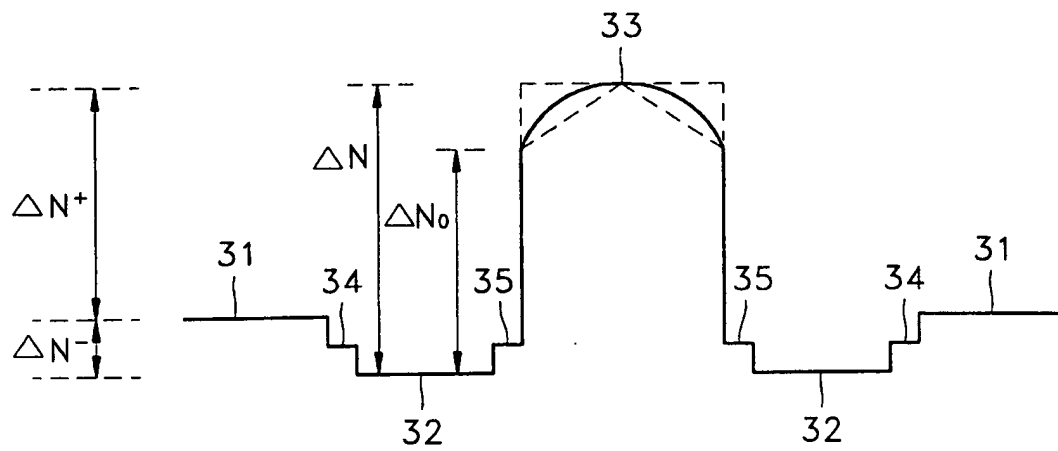
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FIG. 2



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FIG. 3



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FIG. 4A

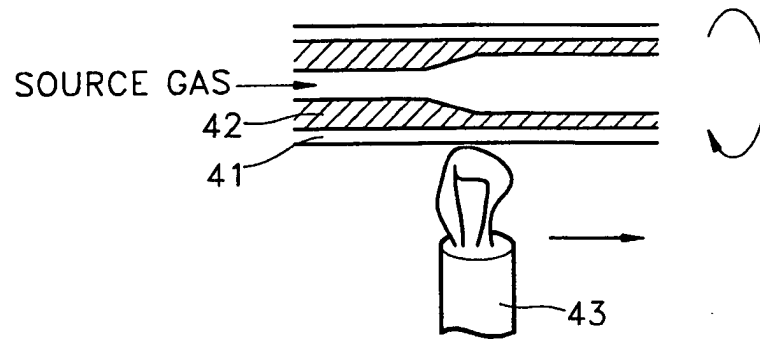


FIG. 4B

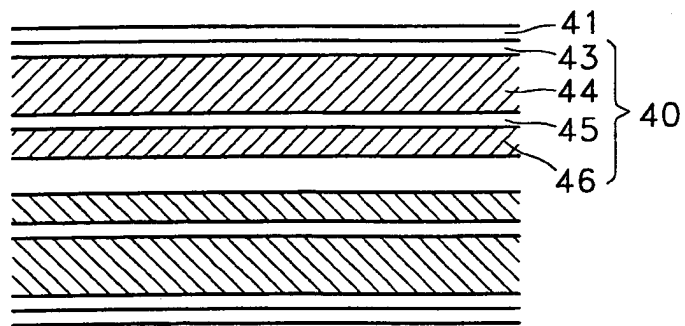
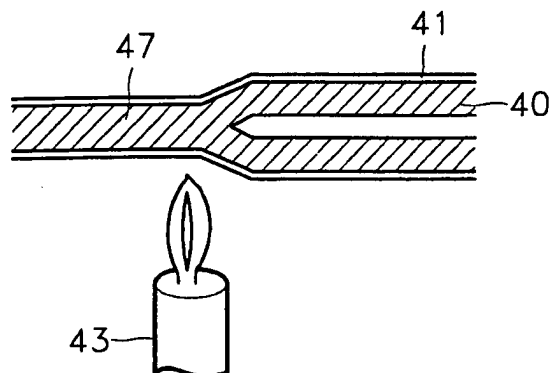


FIG. 4C



## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/KR 99/00329

## A. CLASSIFICATION OF SUBJECT MATTER

IPC<sup>6</sup>: Int.Cl.: C 03 B 37/018, C 03 C 13/04

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC<sup>6</sup>: C 03 B, C 03 C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 56 014430 A (NIPPON DENSHIN DENWA KOSHA). Patent Abstracts of Japan, Vol. 5, No. 62, (C 52) 25 April 1981 (25.04.81).	1, 12
A	JP 55 016244 A (FURUKAWA ELECTRIC CO LTD.) 17 December 1980 (17.12.80), (abstract) [Online] [retrieved on 28 September 1999 (28.09.99)] Retrieved from: EPO WPI Database.	1, 12
A	JP 56 114840 A (HITACHI DENSEN K.K.). Patent Abstracts of Japan, Vol. 5, No. (C 82) 10 December 1981 (10.12.81).	1, 12
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☐ Further documents are listed in the continuation of Box C.☒ See patent family annex.

## \* Special categories of cited documents:

„A“ document defining the general state of the art which is not considered to be of particular relevance

„E“ earlier application or patent but published on or after the international filing date

„L“ document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

„O“ document referring to an oral disclosure, use, exhibition or other means

„P“ document published prior to the international filing date but later than the priority date claimed

„T“ later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

„X“ document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

„Y“ document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

„&amp;“ document member of the same patent family

Date of the actual completion of the international search

28 September 1999 (28.09.99)

Date of mailing of the international search report

08 November 1999 (08.11.99)

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

I . national application No.  
PCT/KR 99/00329

In Recherchenbericht angeführtes Patentdokument Patent document cited in search report Document de brevet cité dans le rapport de recherche	Datum der Veröffentlichung Publication date Date de publication	Mitglied(er) der Patentfamilie Patent family members Membre(s) de la famille de brevets	Datum der Veröffentlichung Publication date Date de publication
JP A2 55162440	17-12-1980	keine - none - rien	